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A multi-stage triaxial testing procedure for low permeable geomaterials applied to Opalinus Clay

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ABSTRACT

In many engineering applications, it is important to determine both effective rock properties and the rock behavior which are representative for the problem's in situ conditions. For this purpose, rock samples are usually extracted from the ground and brought to the laboratory to perform laboratory experiments such as consolidated undrained (CU) triaxial tests. For low permeable geomaterials such as clay shales, core extraction, handling, storage, and specimen preparation can lead to a reduction in the degree of saturation and the effective stress state in the specimen prior to testing remains uncertain. Related changes in structure and the effect of capillary pressure can alter the properties of the specimen and affect the reliability of the test results. A careful testing procedure including back-saturation, consolidation and adequate shearing of the specimen, however, can overcome these issues. Although substantial effort has been devoted during the past decades to the establishment of a testing procedure for low permeable geomaterials, no consistent protocol can be found. With a special focus on CU tests on Opalinus Clay, this study gives a review of the theoretical concepts necessary for planning and validating the results during the individual testing stages (saturation, consolidation, and shearing). The discussed tests protocol is further applied to a series of specimens of Opalinus Clay to illustrate its applicability and highlight the key aspects.

1. Introduction

In many engineering applications, such as nuclear waste repository design, conventional and unconventional oil and gas extraction, and CO_2 sequestration, it is of great interest to assess short- and long-term performances of underground structures like wellbores, repository drifts, and caverns. This requires the determination of effective rock properties and the rock behavior which are representative for the problem's in situ conditions. In both nuclear waste repository design and oil and gas industry, low permeable argillaceous rocks, especially clay shales, are frequently encountered. To quantify the effective strength and to understand the deformation behavior of a clay shale, test specimens often are extracted from the ground and brought to the laboratory. During this process, the samples will undergo a complex stress path and may be exposed to atmospheric conditions. Because of the low permeability of clay shales and usually high drilling and extraction rates, the sampling procedure can be considered as undrained (Anagnostou and Kovári, 1996). Therefore, pore water pressure within the sample will drop due to unloading. In an ideal case of sampling (assuming a homogeneous, isotropic elastic, saturated material with a compressibility of the rock matrix which is much lower than that of water), the pore pressure will drop, according to Skempton (1954), by the same amount as the mean stress changes and the mean effective stress within the sample remains unchanged (i.e. it stays equal to the in situ conditions). Clay shales, however, exhibit a non-isotropic material behavior and therefore the mean effective stress within the extracted samples is likely not comparable to in situ conditions (Skempton and Sowa, 1963; Okumura, 1971; Schjetne, 1971; Graham et al., 1987, 1990; Doran

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et al., 2000). In addition, there are various processes that can lead to a further modification of the effective stress such as desaturation due to gas escaping from solution upon unloading, air-entry and capillary effects due to contact with air and desaturation by cavitation (Okumura, 1971; Young et al., 1983; Hight, 2003; Pei, 2003; Ewy, 2015). Furthermore, the degree of saturation and water content of the samples may further change during storage, core-dismantling and specimen preparation (Monfared et al., 2011; Ewy, 2015; Wild et al., 2015a).

The change from the in situ stress state to the new stress state in the laboratory can affect the representativeness of the measurements. Once the degree of saturation of the specimen drops below 100%, the effective stress law for saturated porous media is no longer valid (Jennings and Burland, 1962; Bishop and Blight, 1963) and the effective stress in the specimen prior to testing remains unknown. Furthermore, the degree of saturation may change during triaxial testing as a consequence of specimen compaction and dilation, which affects the reliability of the test results (Lowe and Johnson, 1960; Bishop and Henkel, 1962; Bishop and Blight, 1963). The stress change during sample extraction and specimen preparation can also directly alter the properties of the specimens. The effect of capillary pressures on the mechanical properties such as strength and deformability has been demonstrated by various researchers (e.g. Fredlund et al., 1978; Schmitt et al., 1994; West, 1994; Ramos da Silva et al., 2008; Wild et al., 2015a). An increase in strength or stiffness with increasing suction has been consistently observed. Additionally, the change in stress from ground to laboratory can cause changes in the structure of the specimen and thus create properties different to the ones in situ (Graham et al., 1990).

Substantial effort has been devoted during the past decades to the establishment of a testing procedure for low permeable soils and rocks (e.g. Lowe and Johnson, 1960; Bishop and Henkel, 1962; Wissa, 1969; Bellwald, 1990; Steiger and Leung, 1991a, b; Aristorenas, 1992; Head, 1998; Barla, 1999; Vogelhuber, 2007; Dong et al., 2013). However, different procedures have been applied during testing. Some researchers (e.g. Steiger and Leung, 1989, 1991a, 1992; Horseman et al., 1993; Horsrud et al., 1994, 1998; Ewy et al., 2003; Islam and Skalle, 2013) conducted tests comprising three steps: (1) loading to a predetermined level of pore pressure and confining pressure, (2) consolidation of the specimens, and (3) axial loading at constant axial strain/displacement rates. In those tests, saturation has been achieved through consolidation but has not been explicitly confirmed. In some of these tests, the specimens have initially been placed into a desiccator to equilibrate with a constant level of relative humidity and thus achieve a specific water content (e.g. Chiu et al., 1983; Steiger and Leung, 1991b; Ewy et al., 2003). Other researchers additionally include a saturation phase at the beginning of the tests utilizing back pressures (e.g. Chiu et al., 1983; Bellwald, 1990; Aristorenas, 1992; Taylor and Coop, 1993; Barla, 1999; Deng et al., 2011; Yu et al., 2012; Dong et al., 2013; Bésuelle et al., 2013; VandenBerge et al., 2014). In some studies, the saturation of the specimens has been confirmed by measuring Skempton's pore pressure coefficient B (Skempton, 1954; Baracos et al., 1980; Bellwald, 1990; Wu, 1991; Aristorenas, 1992; Taylor and Coop, 1993; Barla, 1999; Yu et al., 2012; Dong et al., 2013; VandenBerge et al., 2014). A specimen has been assumed to be saturated when the B-value was higher than a certain value or constant for subsequent measurements. Others considered a specimen to be saturated when the fluxes of water stabilized (Bésuelle et al., 2013) or the pore pressure at the outlet and inlet equilibrated (Wu et al., 1997; Hu et al., 2014).

Depending on the permeability and size of the specimen, different times ranging from several hours to several days have been allocated for consolidation. Pore pressure changes and strains have been used to confirm complete consolidation of the specimens (e.g. Wu, 1991; Taylor and Coop, 1993; Amorosi and Rampello, 2007). The reported axial strain rates for consolidated undrained (CU) tests also cover a wide range of values from the order of $10^{-8}\,s^{-1}$ (e.g. Steiger and Leung, 1991a) to $10^{-4}\,s^{-1}$ (e.g. Graham and Li, 1985; Marsden et al., 1992).

This paper elaborates, based on theoretical considerations from literature, a testing procedure for CU triaxial tests for Opalinus Clay, which is a Mesozoic clay shale chosen as host rock for a nuclear waste repository in Switzerland (BFE, 2011). At the same time, the paper aims at giving an overview of theoretical concepts for planning tests on low permeable materials. The described testing procedure is further applied to a series of Opalinus Clay specimens. Test conditions that allow for testing properties and behavior of Opalinus Clay relevant to the evaluation of tunnel construction at the Mont Terri underground rock laboratory (URL) are chosen. Results are presented and discussed to illustrate the applicability of the proposed laboratory protocol for low permeable clay shales and highlight the key aspects that have to be considered during the individual stages (i.e. saturation, consolidation, and shearing). The interpretation and discussion of the results with respect to the strength and properties are presented in another paper of the authors.

2. Theoretical background and testing procedure

2.1. Saturation stage

2.1.1. Theoretical considerations on the back pressure needed to establish saturation

To avoid unnecessary swelling due to contact with water during the setup, the dry setting method, which does not allow the specimen to take up water during the setting, is preferred (Lo Presti et al., 1999). However, this setting method requires a flushing phase as a preparatory phase for complete specimen to achieve saturation of the pore pressure lines. Using de-aired water avoids bringing additional gas into the system (i.e. pressure lines and pore space). Furthermore, the use of pore water with a composition similar to the in situ pore water is recommended since clay shales are prone to chemical reactions that may alter the geomechanical properties (Ewy et al., 2008). This is especially important for long-term tests in order to keep the influence of the pore fluid purely mechanical.

A small pressure gradient is applied between the bottom (inlet) and the top pore pressure circuit (outlet) by leaving the exit valve open (Barla, 2008). This allows gas to escape from the pore space and from the circuit as pore water permeates the specimen. A confining pressure which exceeds the pore pressure within the specimen and is large enough to minimize swelling and associated damage of the clay shale structure and diagenetic bonds (i.e. degradation of diagenetic bonds) is mandatory (Barla and Barla, 2001; Barla, 2008; Wild et al., 2015b). The effective confining pressure required to minimize swelling during the flushing phase could be determined in the pre-test. Thereby, the confining pressure is increased until swelling is negligible. The determined effective confining pressure can be applied to the subsequent tests.

The actual saturation procedure requires an increase of back pressure at the specimen's faces. This decreases the volume of trapped gas bubbles according to Boyle's law, which reduces the required time to dissolve the gas (Lee and Black, 1972). At the same time, the amount of air which is soluble in water increases according to Henry's law (Lowe and Johnson, 1960). Theoretical relationships between the initial degree of saturation and the required change in back pressure necessary to completely saturate a specimen considering Henry's law have been given by Bishop and Eldin (1950) and Lowe and Johnson (1960).

The back pressure is typically increased in several stages on both specimen faces and is maintained for several hours to days (Lowe and Johnson, 1960; Bishop and Henkel, 1962; Wissa, 1969). The confining pressure is increased simultaneously in such a way as to maintain the effective stress that has been established during the flushing phase.

2.1.2. Demonstration of saturation and validity of Skempton's pore pressure parameter B

Skempton's pore pressure coefficient B can be determined between each back pressure stage (so called B-check) and can be used to ensure saturation of the specimen during the saturation stage. The parameter B represents the ratio between a change in pore pressure and a change in confining pressure (under undrained conditions) (Skempton, 1954). Its value (between 0 and 1) is dependent on the porosity (n), the compressibility of the skeleton (c_d), the compressibility of the fluid (c_f), and the compressibility of the solid material (c_s) (Bishop, 1966):

$$B = \frac{1}{1 + n\frac{c_f - c_s}{c_d - c_s}}$$

Gas bubbles in the pore water system or in the specimen will increase the compressibility of the pore fluid and therefore decrease B. For an ideal, isotropic porous media, with a compressibility of the mineral skeleton greater than 10^{-7} Pa^{-1}, B equals unity when the specimen is saturated (Wissa, 1969). For many rocks and soils, and especially for clay shales, B can be significantly smaller than unity since the load is partly taken by the rock skeleton and the compressibility of the pore fluid is comparable to the compressibility of the rock skeleton (Skempton, 1954; Wissa, 1969). Furthermore, the value of B is dependent on the effective confining pressure that affects the compressibility of the rock skeleton. A decrease in B with increasing effective confining pressure has widely been observed for various rock types such as sandstone, limestone, marble, granite (e.g. Mesri et al., 1976; Green and Wang, 1986; Hart and Wang, 1999; Lockner and Stanchits, 2002), and shales (Mesri et al., 1976; Cook, 1999; Hart and Wang, 1999; Wild et al., 2015b). Additionally, the value of B depends on the compliance of the testing system (Wissa, 1969; Bishop, 1976). Pore pressure lines, transducers, and Darcy filters (porous plates) influence the compliance and add porosity to the system. The more compliant the system, the smaller the excess pore pressure that is measured, which decreases the B-value (Wissa, 1969; Monfared et al., 2011; Hu et al., 2014). An undrained compression tests may also lead to an instantaneous pore pressure change measured outside the specimen that is different from the pore pressure change in the specimen (Monfared et al., 2011). In case where the effective volume of the external system is comparable to the pore volume of the specimen, the assumption of an undrained response is not valid anymore (Bishop, 1973; Ghabezloo and Sulem, 2010; Monfared et al., 2011; Hu et al., 2014). A very rigid external system with a small free volume is therefore crucial. Correction calculations to account for the effects of the system are given by different authors (e.g. Bishop, 1976; Bellwald, 1990; Ghabezloo and Sulem, 2010; Monfared et al., 2011).

For the reasons stated above, it is not sufficient to target a high B-value (e.g. higher than 0.95) as a standalone criterion for demonstrating complete saturation in low permeable clay shales. A high value of B is, however, an indicator for a high degree of saturation.

Due to a high effective confining pressure, a high compressibility of the system or rock skeleton, B may be smaller (e.g. 0.8) although the specimen is saturated. Wissa (1969) noted that for low permeable rocks, B will remain constant for two subsequent undrained confining pressure changes if the specimen is completely saturated (i.e. all compressible gas bubbles have been dissolved in the pore water). Aristorenas (1992) stated that it is almost impossible to reach identical Skempton's pore pressure coefficient B from subsequent B-checks. Therefore, he assumed a specimen to be saturated if B does not change significantly (ΔB in the order of ± 0.03) for two subsequent steps. Hence, for demonstrating full saturation, the absolute value of B and the assessment of the change between two subsequent B-checks can be used.

2.2. Consolidation stage

Subsequent to the saturation stage, a consolidation stage is used to establish the desired value of effective stress within the specimen prior to shearing. Pore pressure and confining pressure will be maintained at the chosen values. The pore pressure valves on one side or on both sides are opened and the specimen is allowed to consolidate against a back pressure which is equal to or higher than the back pressure applied during the saturation phase in order to maintain full saturation. Complete consolidation is essential to measuring adequate pore pressure values as insufficient consolidation may lead to an overestimation of the pore pressure and thus an underestimation of the effective strength.

The time theoretically required to consolidate a specimen can be estimated prior to testing based on the one-dimensional (1D) consolidation theory by Terzaghi (1943). The time needed to dissipate excess pore pressure is dependent on the square of the drainage length and the calculation strongly relies on the coefficient of consolidation. The coefficient of consolidation for an isotropic consolidation in a triaxial cell, however, is not the same as that for a 1D consolidation as used in Terzaghi's 1D consolidation theory (Gibson and Henkel, 1954; Bishop and Henkel, 1962; Head, 1998). An approximation for derivation of the coefficient of isotropic consolidation is given by Head (1998). Alternatively, the coefficient of (isotropic) consolidation can be estimated from experimentally determined time–settlement curves using different approaches (e.g. Bishop and Henkel, 1962; Robinson and Allam, 1996; Head, 1998; Germaine and Germaine, 2009).

For an isotropic consolidation in a triaxial cell, where the stress is applied from all three directions equally, the specimen consolidates in three dimensions. Therefore, strictly speaking, the three-dimensional (3D) theory of consolidation applies. If only vertical drainage via the porous stones at top and bottom of the specimen is permitted, Terzaghi's 1D consolidation can be applied as an adequate approximation for the time required for full consolidation (Scott, 1963). Filter strips used at the side of the specimen might decrease the time required for full consolidation by allowing radial drainage and thus shorten the drainage length (Bishop and Henkel, 1962; Leroueil et al., 1988; Mitachi et al., 1988). If both vertical and radial drainage is allowed and the specimen height is assumed to be twice its diameter, a combination of the solution for radial and vertical drainage can be used (Gibson and Lumb, 1953; Scott, 1963).

In practice, the degree of consolidation is usually controlled by examining time-dependent variations in volumetric strain. Furthermore, the change in water content due to excess pore pressure dissipation can be analyzed. The consolidation of the specimen is considered sufficient when the strain approaches a constant value and the water content remains constant.

2.3. Shearing stage (undrained shearing)

2.3.1. Theoretical considerations on the loading/strain rate

In principle, the load during undrained shearing can be applied substantially fast. However, to measure the excess pore pressure close to the end faces of the specimens, a sufficiently slow loading/strain rate is required that allows for a redistribution of pore

pressure within the specimen in order to measure a pore pressure representative for the bulk specimen (which is necessary to determine the effective strength). Clay shales tend to compact or dilate during shearing, causing the pore pressure to change. Pore pressure transducers are normally connected via filter material and drainage lines close to the specimen's top and/or bottom end faces. Due to their compliance, minor volume changes are possible, causing a small fluid flow in or out of the specimen (Bishop and Henkel, 1962; Wissa, 1969; Monfared et al., 2011; Hu et al., 2014). This is necessary to achieve pore pressure equalization between the specimen and the drainage system. The time needed for this equalization depends on the compliance of the drainage system. The higher the compressibility of a system, the longer it takes to redistribute pore pressure changes induced by shearing and hence the loading/strain rate has to be reduced to measure pore pressures representative for the bulk behavior of the specimen (Whitman et al., 1961; Wissa, 1969; Monfared et al., 2011). Furthermore, in a standard triaxial compression test, the pore pressure at the ends of the specimen will be slightly higher than that at its center due to unequal loading caused by boundary effects (Blight, 1963; Peng, 1971). The equalization of the pore pressure within the specimen additionally depends on its permeability and dimensions (Bishop and Henkel, 1962). From experimental test results of different shales and clays, Blight (1963) concluded that for an overconsolidation ratio up to 20, a degree of pore pressure equalization of 95% is sufficient to avoid an error in effective confining pressure larger than 5%. Theoretical relationships for the time to failure for CU tests are given by Gibson (Bishop and Henkel, 1962). The time required to reach the peak strength can further be used to determine an appropriate loading/strain rate when the load or strain at failure is known (Bishop and Henkel, 1962). If not known in advance (e.g. from previous existing published data), this parameter adds an uncertainty to the estimation of the appropriate loading/strain rate, in addition to the uncertainties in estimating the time required to equalize nonuniform pore pressure within the specimen.

2.3.2. Practical considerations on the loading/strain rate

The above considerations are solely based on theoretical considerations and uncertainties remain in the choice of an adequate loading/strain rate. A more robust, but very time-consuming way to derive an appropriate loading/strain rate for CU tests is a series of triaxial tests which utilizes rates that vary one order to two orders of magnitude (e.g. 10^{-6} s^{-1}, 10^{-7} s^{-1}, and 10^{-8} s^{-1}). Such a test series requires full saturation of the specimens. Furthermore, the material characteristics of different specimens used for evaluation should be comparable. The loading/strain rate is adequate for CU tests on specimens of similar test material and with similar dimensions if the pore pressure response does not change between two tests that utilize different loading rates.

To check if the loading/strain rate for a CU test is appropriate, pore pressure magnitudes that evolve during elastic shearing can be examined using Skempton's pore pressure coefficients *A* and *B* (Skempton, 1954). For an isotropic, perfectly elastic material, *A* equals 1/3 (Skempton, 1954). For an anisotropic material, *A* will be smaller or larger than 1/3 depending on the orientation of the applied change in axial stress with respect to the anisotropy. Note that *A* also depends on the magnitude of the applied stress and is often reported as *A* at failure. This is not the case in Skempton and Bjerrum (1957) who reported *A*-values for overconsolidated clays that are representative for the elastic response. Values between 0.25 and 0.5 are given. *B* can be taken at the end of the saturation phase (i.e. on a saturated specimen) or calculated based on the relations given by Bishop (1973). However, depending on the confining pressure used during triaxial shearing, the dependence of *B* on the effective confining pressure needs to be considered. Using these theoretical values for *A* and *B* (or using the measured value for *B*), a theoretical value *AB* can be calculated and used as an indicator for a correct loading/strain rate. If the measured *AB* is significantly lower than the theoretical value, the loading/strain rate is too fast to capture the actual pore pressure response of the specimen. Note that this criterion can only be used if the saturation phase and the consolidation phase are complete. Incomplete saturation or nonuniform distribution of pore pressure during shearing would reduce the *AB*-value substantially.

3. Application of the testing procedure to Opalinus Clay

3.1. Material description

Examples of CU tests conducted on Opalinus Clay are taken to illustrate the applicability of the testing procedure afore-described and highlight the aspects that have to be considered when testing low permeable clay shales. Opalinus Clay was deposited in a shallow marine environment about 180 million years ago. The samples used for this study have been cored in the shaly facies at the Mont Terri URL in Switzerland (Wild, 2016). The main mineralogical constituent of the shaly facies at the Mont Terri URL are clay minerals (30%–80%), quartz (10%–30%), carbonates (5%–20%), and feldspar (0–5%) (Thury and Bossart, 1999; Bossart, 2005; Klinkenberg et al., 2009). The recent overburden at the Mont Terri URL is about 250 m but it is estimated to have reached about 1350 m in the past (Mazurek et al., 2006). Opalinus Clay can therefore be considered as overconsolidated. Due to its complex history of sedimentation, burial, physical compaction, development of diagenetic bonding, tectonic faulting, uplift, and erosion, Opalinus Clay shows a pronounced bedding (Van Loon et al., 2004; Marschall et al., 2005). Its physical behavior is therefore often considered as transversely isotropic. The hydraulic conductivity varies between the order of 10^{-12} m/s and 10^{-14} m/s depending on the orientation with respect to bedding and the confining pressure (Marschall et al., 2004). The water loss porosity (calculated from weight loss at 105 °C and grain density) varies between 12% and 18% (Thury and Bossart, 1999).

3.2. Test conditions and stress state at the Mont Terri URL

Test conditions were used which mostly resembled the conditions at the Mont Terri URL and therefore allowed for the measurement of properties and behavior that can be used for the performance assessment of excavations at this location. The in situ stress state at the Mont Terri URL has been studied intensively in the past two decades utilizing different approaches. Results from 3D numerical models, stress-induced borehole break-outs, undercoring, borehole slotter, and hydraulic fracturing tests have been compared by Martin and Lanyon (2003). It is shown that stress measurements in a transversely isotropic material such as Opalinus Clay are challenging and especially the magnitude of the minimum principal stress is difficult to determine (Evans et al., 1999; Martin and Lanyon, 2003; Corkum, 2006). Despite the uncertainties in the minimum principal stress at the Mont Terri URL, it is assumed here that the magnitudes are reasonable well constrained. The components of the considered stress tensor are given in Table 1.

Table 1
In situ stress state considered for the determination of the test conditions for the CU tests (MPa).

σ_1	σ_2	σ_3
6.5	4.5	2.5

Note: σ_1, σ_2 and σ_3 are the maximum, intermediate and minimum principal stresses, respectively.

Table 2
Water content of core pieces tested directly after drilling.

Sample no.	Depth (m)	Water content (%)
1	2.4	7.5
2	2.45	7.5
3	8.5	7
4	12.35	7.4
5	12.35	7.3
6	~17.7	5.3
7	~20.4	8.1

Additionally, a pore pressure of 2 MPa is considered (except for specimens ETH16 and ETH17 for which a pore pressure of around 1 MPa was used). Although the in situ stress tensor seems to be strongly anisotropic, isotropic conditions were used for simplicity reasons within this study. Effective stresses between 0.5 MPa and 16 MPa were considered to be relevant for the assessment of tunnel construction at the Mont Terri URL.

3.3. Sample extraction, specimen preparation and setup

3.3.1. Core sampling

The samples were taken from two boreholes (BHM-1 and BHM-2) with a core diameter of 67.5 mm. The 25 m long boreholes were oriented parallel (BHM-1) and normal (BHM-2) to bedding. Triple tube core barrel technique with pressurized air cooling was used to obtain high quality cores. Small core pieces of BHM-1 at different depths were used to determine the water content of the core samples after drilling and core extraction. The water content was determined according to International Society for Rock Mechanics (ISRM) suggested methods (ISRM, 1979). The individual water contents are listed in Table 2. They are, except for one sample, consistent with the water content reported in the literature (6%–8.6%, e.g. Pearson et al., 2003).

3.3.2. Sample storage and preparation

The cores were covered by a plastic tube, sealed immediately after core extraction in vacuum-evacuated aluminum foil, and stored in wooden boxes. After arrival in the laboratory, the cores were stored at constant humidity and temperature. To avoid undesired desaturation, which is often accompanied by desiccation cracks (Wild et al., 2015a), the specimen preparation procedure was optimized and reduced to 20–30 min. Smooth and precise cutting of the sub-sample into specimens with a length of about 135 mm was obtained by using a diamond band saw (Proxxon, Model MBS 240/E) that operates with pressurized air instead of water cooling. The feed rate was manually controlled. A two-trail system was designed and manufactured to allow for cutting the edges of the specimen along a planar surface, perpendicular to the core axis. Two different orientations of specimens were distinguished: P-specimens, where the bedding is aligned with the core axis, and S-specimens, where the bedding is aligned normal to the core axis.

After preparation, the specimen was measured, weighted, photographed, and placed in the triaxial cell using the dry setting method. Eight strips and two circles of filter paper were placed on the specimen's side and top/bottom faces, respectively, as shown in Fig. 1a before a rubber membrane (2 mm thick) was put over the specimen. O-rings were put in place for appropriate sealing at the top cap and at the pedestal while the inlet and outlet at the top and bottom were closed to avoid air entering the specimen. At this point, the specimen was sealed and isolated from the laboratory environment and the following steps (mounting local displacement transducers, connecting pore pressure circuit, closing the pressure vessel, etc.) could be performed (Fig. 1b).

According to Bishop and Henkel (1962), there is an effect of the rubber membrane and the side drains on strength. However, when the specimen fails at low values of strain (i.e. below 4%–5%), Bishop and Henkel (1962) suggested that a combined membrane and side drain correction of about 13 kPa is sufficient. This is the case for Opalinus Clay (Amann et al., 2011, 2012; Wild, 2016) and compared

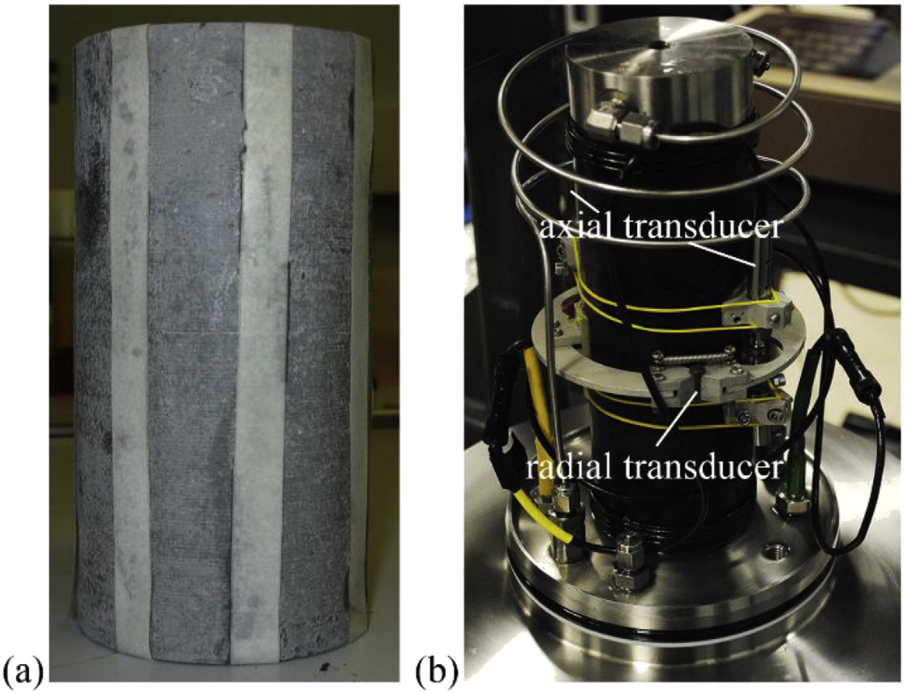

Fig. 1. (a) Specimen prepared with filter paper strips at lateral sides; (b) Specimen setup in the triaxial cell with pore pressure line and axial and radial transducers to measure axial and radial displacements locally.

Table 3
Properties and initial test conditions (total confining pressure and pore pressure after consolidation) of specimens.

Specimen no.	Specimen orientation	Diameter (mm)	Height (mm)	Water content before test (%)	Dry density (g/cm^3)	Porosity (%)	Degree of saturation before test (%)	Confining pressure (MPa)	Initial pore pressure (MPa)
ETH08	S	67.68	123.74	7.5	2.26	17.2	98.6	3.09	2.09
ETH09	S	67.74	135.88	7.5	2.27	17.1	98.9	4.09	2.1
ETH10_2	S	67.59	133.89	7.4	2.27	17	98.9	6.09	2.06
ETH16	P	67.47	133	7	2.28	16.6	96.2	1.6	1.11
ETH17	P	67.47	135.35	6.4	2.27	16.8	86.6	1.69	0.95
ETH19	P	67.5	134.4	6.1	2.28	16.6	83.4	4.08	2.09
ETH20_2	P	67.65	133.3	7.2	2.26	17.5	92.7	6.08	2.1
ETH21	P	67.56	133.99	7.1	2.26	17.3	92.9	8.09	2.06
ETH22	P	67.45	134.31	7	2.26	17.1	92.2	10.08	2.11
ETH23	P	67.45	133.81	6.8	2.29	16.3	94.9	14.1	2.1
ETH24	P	67.43	134.11	7	2.26	17.4	91.1	18.08	2.1

to its relatively high peak strength (i.e. 3–29 MPa differential stress, depending on the confining pressure (Wild, 2016)), this influence is negligible.

3.3.3. *Water content and degree of saturation*

The water content and degree of saturation determined after sample extraction (Table 2) was compared to the values determined after specimen preparation (Table 3) to quantify any severe desaturation that may have taken place during specimen preparation. The water content given in Table 3 was determined with respect to the weight of the specimens after 24 h drying at 105 °C. Specimens ETH10_2 and ETH20_2-ETH24 were dried to constant weight. The comparison between the water content calculated after 24 h drying and after drying to constant weight (reached after about 2 d) for these specimens revealed that the water content after 24 h underestimates the water content by about 0.4%. Similar results have been observed for specimens tested by Amann et al. (2011, 2012). It can be seen that most specimens show a water content that lies slightly below the values measured after drilling but within the variability reported in the literature (6%–8.6%, e.g. Pearson et al., 2003). The water content changed even though the preparation procedure was optimized and required only 20–30 min. This decrease in water content might be significant in terms of strength/stiffness (Wild et al., 2015a).

Also given in Table 3 are the dry density, porosity, and degree of saturation before the test that were determined according to the ISRM suggested methods (ISRM, 1979). Grain density for Opalinus Clay ranges between 2.69 g/cm^3 and 2.78 g/cm^3 (Pearson et al., 2003; Bossart, 2005; own data). A mean value of 2.73 g/cm^3 was considered in this study to calculate the porosity.

3.3.4. *Triaxial apparatus*

The triaxial tests in this study were conducted at the DIPLAB Geomeccanica Laboratory of the Politecnico di Torino (Italy) using two triaxial apparatus (HPTA, high pressure triaxial apparatus and MPTA, medium pressure triaxial apparatus) which were manufactured by GDS Instruments Ltd. and modified in the laboratory (Barla et al., 2010).

Axial load/displacement, radial pressure/displacement and back pressure/volume applied to the specimen can be controlled individually. The two machines differ in the maximum possible confining pressure/load/back pressure that can be applied. For the MPTA machine, confining pressures up to 20 MPa, back pressures up to 16 MPa, and axial loads to maximum of 64 kN can be applied. For the HPTA machine, confining pressures up to 64 MPa, back pressure up to 20 MPa, and deviatoric axial loads up to 250 kN are possible. The axial displacement is determined externally with an accuracy of 1 μm by measuring the displacement of the sliding plate of the loading frame. Additionally, radial and axial displacements were measured locally by a set of linear variable differential transformers (LVDTs) (i.e. two axial transducers which are diametrically opposed and a radial transducer which is mounted on a belt at half the specimen's height) that were glued on the membrane (Fig. 1b). The accuracy of the vertical measurements is 1 μm on a full scale of 10 mm, and the accuracy of the radial measurement is 0.5 μm on a full scale of 5 mm. The load can be applied and measured with a load cell placed between the upper horizontal beam of the loading frame and the top cap (accuracy of 60 N). Pore pressure changes can be measured by a transducer located in the back pressure controller, which is connected to the bottom end of the pore pressure circuit, and by an external transducer which is directly connected to the top end of the pore pressure circuit, immediately after its exit from the triaxial cell (accuracy of 8 kPa).

4. Assessment of the test results

4.1. *Saturation stage*

De-aired water with composition similar to the in situ pore water at Mont Terri URL (according to the recipe by Pearson (2002)) was used. At the inlet, the back pressure was increased in several steps (Fig. 2). Back pressures between 0.11 MPa and 0.35 MPa were utilized. The outlet was at 1 atm. The stresses were increased in

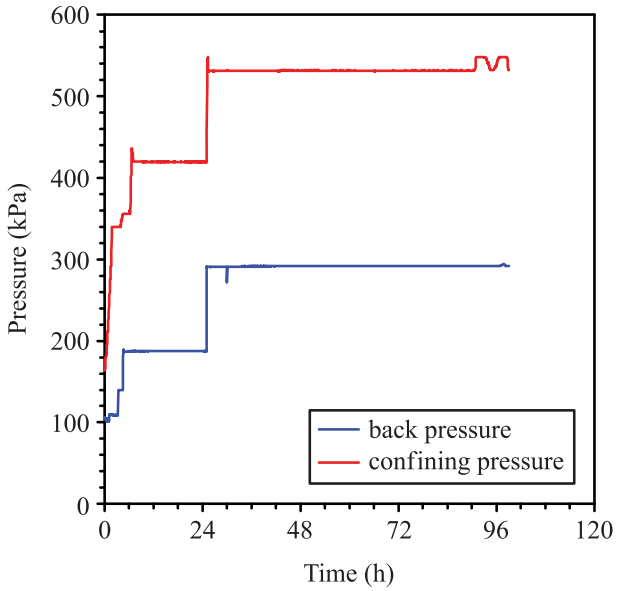

Fig. 2. Example of pressure increase during the flushing phase for test specimen ETH20_2.

several steps. Towards the end of the flushing phase, the outlet was closed for the majority of the specimens until pore pressure equilibration between the inlet and outlet was observed. On average, the duration of the flushing phase was between 1.1 d and 6 d.

Subsequent to the flushing phase, the specimen was further saturated by increasing the back pressure at both specimen's faces. Fig. 3 shows the typical evolution of back pressure and confining pressure during the saturation phase. The back pressure was increased in several steps. Before each back pressure increase, the valves were closed and saturation was checked by performing *B*-checks. Back pressure phases lasted for hours to days whereas equilibration during *B*-checks was reached within about 1–2 h. The whole saturation phase took several days to weeks. The resulting curves for the individual *B*-checks are shown in Fig. 4. A specimen was assumed to be saturated if the *B*-value was sufficiently high and did not change significantly by more than ±0.03 for two subsequent *B*-checks. This criterion is valid for all specimens shown in Fig. 4 except for specimens ETH08 ($\Delta B = 0.08$), ETH10_2 ($\Delta B = 0.05$) and ETH16 ($\Delta B = 0.06$). Although these latter specimens show a change in *B*-value greater than 0.03, the *B*-values itself are sufficiently high (i.e. higher than 0.9). Therefore, specimens ETH08, ETH10_2 and ETH16 are considered to be saturated.

The assessment of the saturation phase based on the test results is in agreement with theoretical considerations dealing with the relationships between the initial degree of saturation and the required change in back pressure necessary to completely saturate the specimens (e.g. Bishop and Eldin, 1950; Lowe and Johnson, 1960). Fig. 5 shows the theoretical curve for the minimum change in back pressure (Δu) needed to saturate a specimen depending on its initial degree of saturation (S_0) according to Lowe and Johnson (1960). Also plotted are the relationships between the initial degrees of saturation (see Table 2) and the maximum changes in back pressure applied to the specimens. The error bars show the saturation range due to the uncertainties in grain density (a value of 2.73 ± 0.03 g/cm^3 was used for calculation here). It can be seen that all specimens were subjected to a back pressure higher than the theoretical pressure necessary to reach saturation.

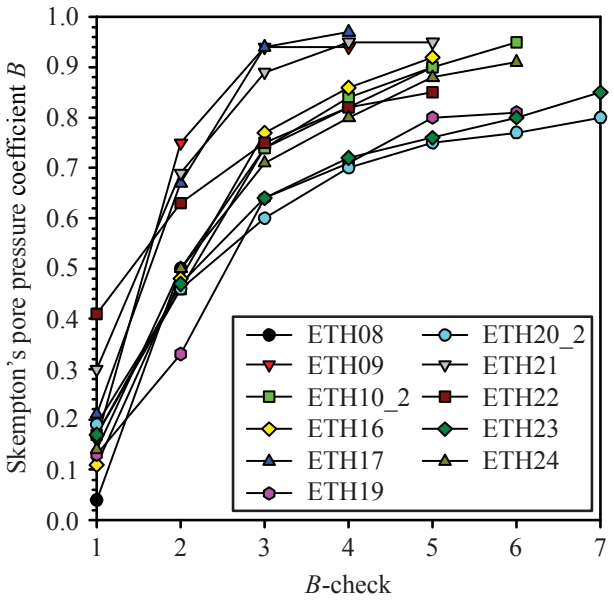

Fig. 4. Values of Skempton's pore pressure coefficient *B* obtained for the individual *B*-checks during the saturation phase.

Swelling during the saturation procedure (i.e. flushing and saturation phases) has the potential to affect the shale structure and damage cohesive bonds in the specimen. Thus, swelling may affect the geomechanical properties derived from triaxial testing (Bjerrum, 1967; Graham and Au, 1984; Calabresi and Scarpelli, 1985; Leroueil and Vaughan, 1990; Takahashi et al., 2005; Picarelli et al., 2006; Cho et al., 2007). It is generally recommended that the saturation phase shall be done under effective stress states similar to the sampling location (Delage et al., 2007; Mohajerani et al., 2011; Monfared et al., 2011; i.e. in our example, 4.5 MPa mean stress and 2 MPa pore pressure) or at the measured residual effective stress after sampling, storage and handling (Cho et al., 2007). Monfared et al. (2011) performed triaxial test on

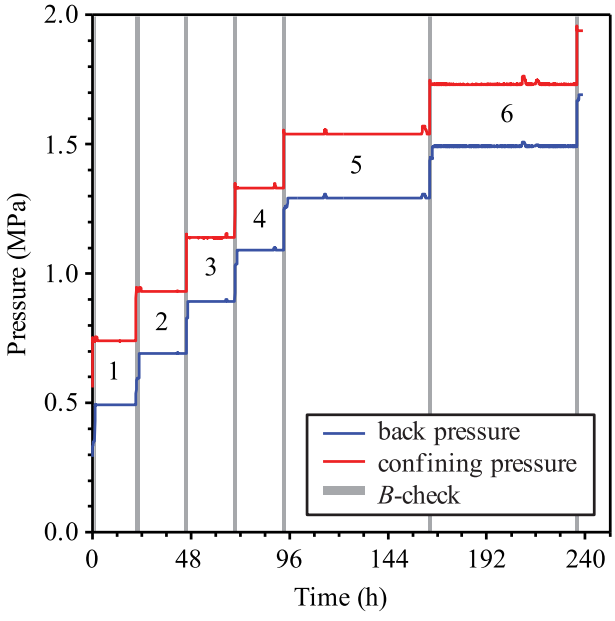

Fig. 3. Example of the saturation phase with its back pressure stages (indicated by numbers) and performed *B*-checks in between (highlighted in gray) for test specimen ETH20_2.

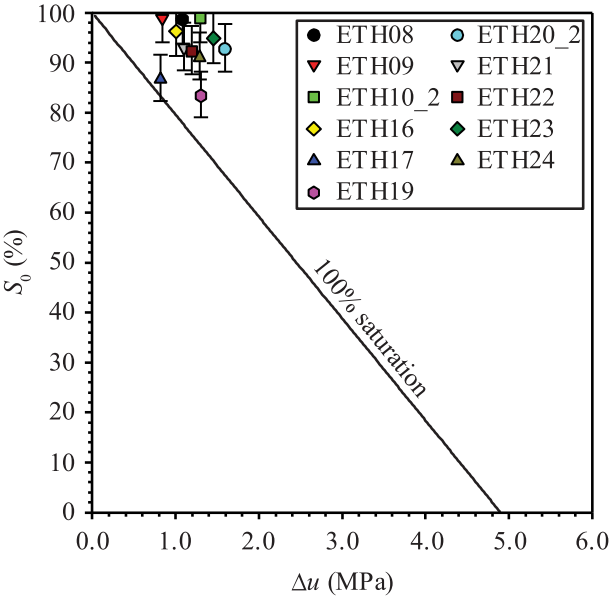

Fig. 5. Back pressure change applied to the individual specimens (symbols) compared to the theoretical pressures required to saturate the specimen to 100% (line) calculated using the relationship given by Lowe and Johnson (1960). The error bars show the saturation range due to uncertainties in grain density.

hollow cylinder specimens of Opalinus Clay which have been saturated at a mean effective stress similar to the Mont Terri URL. The measured volumetric strain during the saturation was around −0.3% after 10 h and swelling continued by a constant swelling rate of -2.02×10^{-5} h^{-1} for a period of time up to 120 h (i.e. 0.54% in total). Menaceur et al. (2015) performed triaxial tests on back-saturated hollow cylinder specimens of Callovo-Oxfordian claystone. Saturation has been established under an effective mean stress of 8 MPa similar to the effective stress state at the Bure URL. The resulting volumetric strains ranged between −1% and −1.2%. Thus, even under effective stresses similar to the sampling location, swelling needs to be anticipated in a range between −0.5% and −1.5% depending on the material type.

The purpose of this study was to establish full saturation in an effective mean stress range lower than in situ (i.e. effective mean stress of 20–600 kPa). This was necessary to avoid unloading and consequently desaturation during the consolidation stage since the tests aimed at consolidation stresses in the range relevant for tunnel construction at the Mont Terri URL (i.e. 0.5–16 MPa). Examples of the measured strains during flushing and saturation are shown in Fig. 6. During the flushing phase (Fig. 6a), the majority of the specimens showed a volumetric strains ranging between −0.3% and −1.1%. During the saturation phase (Fig. 6b), the accumulated volumetric strain at the end of the stage was between −0.1% and −0.8%. Total volumetric swelling strains (i.e. combining the flushing and saturation phases) range between −0.5% and −1.6%. Similar to Monfared et al. (2011), a constant volumetric strain rate ($\Delta\varepsilon_{vol}$) of about -2×10^{-5} h^{-1} was found during the saturation phase (Fig. 6b).

The total volumetric strains that accumulated during flushing and saturation phases are significantly lower than the free swelling strain measured by Horseman et al. (2007), which was −2.9%. Thus, additional damage due to the saturation procedure is considered to be minor.

4.2. Consolidation stage

For consolidation, the confining pressure and back pressure on both end faces of the specimen were increased within 24 h to establish target effective stresses of 0.5 MPa, 0.75 MPa, 1 MPa, 2 MPa, 4 MPa, 6 MPa, 8 MPa, 12 MPa, and 16 MPa. The consolidation stage ranged from 48 h to 163 h in case of P-specimens and from 75 h to 453 h in case of S-specimens. At the end of consolidation, the strain and the change in back volume were constant, indicating complete consolidation. A typical curve of a completely consolidated specimen is shown in Fig. 7.

Ferrari and Laloui (2012) reported values of the coefficient of consolidation from an oedometer test on an S-specimen of Opalinus Clay from the Mont Terri URL, ranging between 0.06 mm^2/s and 0.2 mm^2/s for an effective vertical stress between 1 MPa and 4 MPa. Assuming a Poisson's ratio of 0.2 for Opalinus Clay and applying the relationship proposed by Head (1998), the corresponding coefficient of consolidation for an isotropic consolidation ranges between 0.03 mm^2/s and 0.1 mm^2/s. Using Terzaghi's theory of consolidation

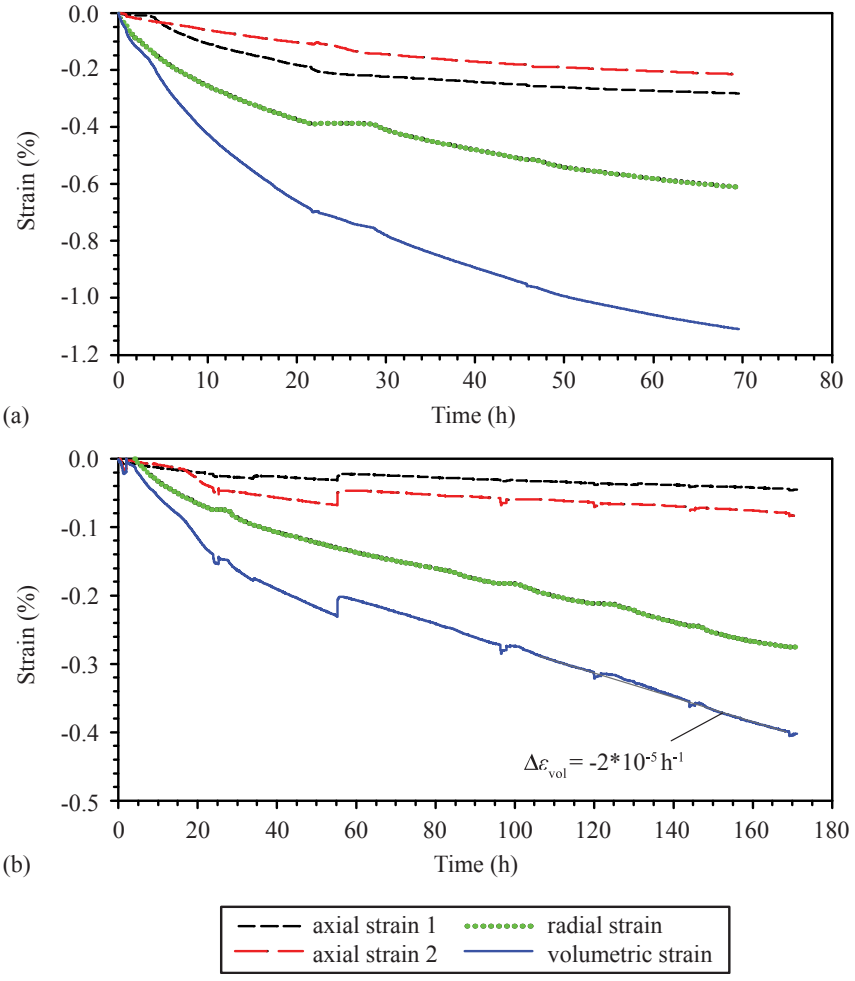

Fig. 6. Typical example (test specimen ETH24) of strains measured during (a) flushing phase and (b) saturation phase.

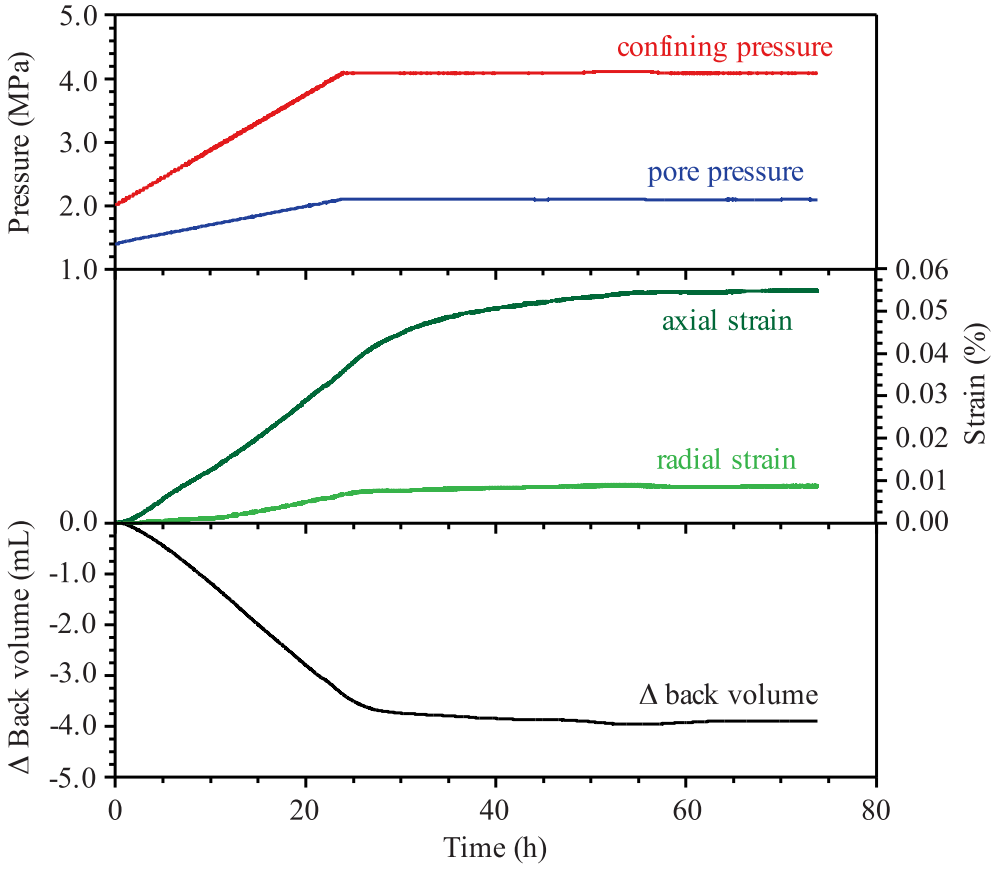

Fig. 7. Example (ETH19) of consolidation stage showing the increases in confining pressure, pore pressure, local axial and radial strains (the two local axial transducers were averaged), and back volume. The change in back volume indicates the change in water content.

(Terzaghi, 1943), the resulting time theoretically required to consolidate a S-specimen is 14–48 h. However, the load applied at the beginning of the consolidation stage is ramped up over a time of 24 h. According to Olson (1977), the required time for consolidation due to ramp loading is estimated to range between 30 h and 68 h. This is less than the actual observed time required for consolidation. The value of the coefficient of consolidation for a P-specimen was unknown prior to testing and therefore no theoretical time could be estimated.

4.3. Shearing stage

CU tests were carried out using constant axial strain rates between 0.88×10^{-6} s^{-1} and 1.25×10^{-6} s^{-1} for P-specimens and between 1.23×10^{-7} s^{-1} and 1.35×10^{-7} s^{-1} for S-specimen. These values lie within the wide range of strain rates for CU tests reported in the literature (i.e. somewhere between 10^{-4} s^{-1} and 10^{-8} s^{-1}, e.g. Graham and Li, 1985; Steiger and Leung, 1991a; Marsden et al., 1992). To check the adequacy of the strain rates, the *AB*-values measured in the elastic region (i.e. at low differential stress) are given in Table 4. A theoretical value was calculated by using the range of *A* given by Skempton and Bjerrum (1957) (i.e. 0.25–0.5) and the measured values for *B* at the end of the saturation stage (i.e. 0.8–0.97). The resulting theoretical *AB*-values expected for fully saturated specimens range between 0.2 and 0.49. The actual measured values *AB* range between 0.15 and 0.69 (Table 4) and are in agreement with the theoretical ones. The chosen axial strain rate can therefore be assumed to be adequate for a CU test.

Table 4
Coefficient *AB* measured for different CU tests.

Specimen no.	*AB*-value
ETH08	0.49
ETH09	0.58
ETH10_2	0.69
ETH16	0.33
ETH17	0.15
ETH19	0.27
ETH20_2	0.26
ETH21	0.2
ETH22	0.22
ETH23	0.24
ETH24	0.22

The strain rates for the S-specimens are also in agreement with theoretical considerations. Assuming a coefficient of consolidation of 0.03–0.1 mm^2/s for S-specimens (see above), an axial strain at peak strength of 0.9%–1.3% (Amann et al., 2012), and using the theoretical relationship to estimate the time to failure for a specimen without lateral drainage given by Gibson (Bishop and Henkel, 1962), an axial strain rate between 0.36×10^{-7} s^{-1} and 1.71×10^{-7} s^{-1} is theoretically required for CU tests on S-specimens. This is in the same range as the axial strain rate for S-specimen (i.e. 1.23×10^{-7}–1.35×10^{-7} s^{-1}) used in this study and is proven to be adequate by assessing the *AB*-value.

5. Conclusions

To overcome the influences of sampling, storage and specimen preparation on the effective properties and behavior of low permeable geomaterials and to acquire reliable parameters

representative for the considered in situ problem (e.g. tunnel excavation), a testing procedure is required that allows the establishment of full saturation, completion of consolidation, and that utilizes a loading/strain rate that is slow enough to capture pore pressure changes during undrained loading. With the focus on excavations in Opalinus Clay at the Mont Terri URL, theoretical concepts of the individual stages of CU tests were presented and their applicability was shown and discussed in a series of test specimens. The main conclusions are drawn as follows:

(1) The water and saturation loss during sampling (including drilling operation and core extraction), storage and specimen preparation can be minimized but not avoided. This unavoidable desaturation is the main reason for the necessity of a laboratory testing procedure that allows to back-saturate the specimen. Measuring water content of samples right after drilling and comparing it to the water content of the specimen after preparation can be used as an indicator of the saturation state of the test specimen.
(2) Saturation within the specimen is established by increasing the back pressure and allowing gas to solve within the pore water. Subsequent tests for Skempton's pore pressure coefficient B (goal: change in B smaller than/equal to approximately 0.03 for two subsequent steps) in combination with a theoretical estimation of the back pressure required to saturate the specimen according to its initial degree of saturation can be used to confirm full saturation.
(3) Swelling during the flushing and saturation phases needs to be anticipated. The use of an effective confining pressure similar to the sampling location may reduce the amount but cannot avoid any swelling and might be impractical in the cases where tests at low effective confining pressures are requested. Total volumetric swelling strains between −0.5% and −1.6% were observed for the specimens tested in this study for effective confining pressures between 20 kPa and 600 kPa. Nevertheless, in comparison to the free swelling strain of −2.9% (Horseman et al., 2007), the measured amount of swelling is small and additional damage due to the saturation procedure was considered to be minor.
(4) Consolidation is used to establish the desired value of effective stress within the specimen prior to shearing. Values of effective stress higher than the ones used during the saturation phase allow the specimen to remain saturated. Factors like loading time, drainage conditions at the boundaries of the specimen (including the effectiveness of filter paper), and the consolidation coefficient significantly influence the estimation of the required consolidation time. Due to uncertainties in estimating the required consolidation time, an assessment of the time-dependent evolution of strains and volume of water flowing out of the specimen provides a more reliable criterion for the completeness of the consolidation phase. The specimen is considered to be sufficiently consolidated if strains and water volume are constant.
(5) The loading/strain rate for CU tests should be sufficiently slow to allow the measurement of pore pressure changes at the end faces of the specimen which are representative for the bulk pore pressure evolution in the specimen during shearing. Estimation of the loading/strain rate prior to testing can be based on the theory of 1D consolidation. Uncertainties in the coefficient of consolidation, however, affect a proper estimation of the loading/strain rate. The measurement of the product of Skempton's pore pressure coefficients AB was used in this study as a data-based assessment criterion for the loading/strain rate.

Conflict of interest

The authors wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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